
Micro-Machined Thin Film H₂ Gas Sensors

**DOE Hydrogen and Fuel Cells
2003 Annual Merit Review**

ATMI

Ing-Shin Chen, Phil Chen, F. DiMeo, Jeff Neuner,
Andreas Rohrl, Michele Stawasz, Jim Welch

Outline

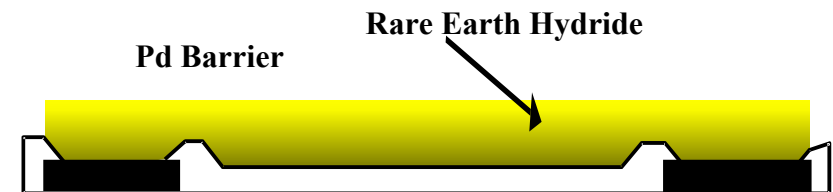
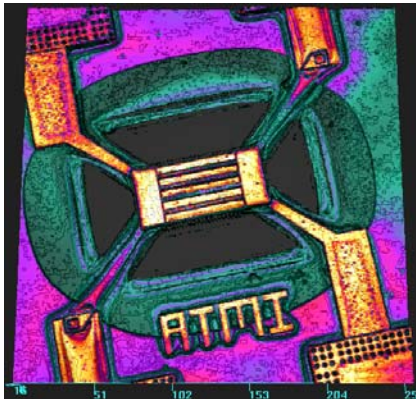
- Introduction
- Approach
- Project Timeline
 - Overall and FY2003
- Accomplishments/Progress
 - Focus on Understanding and Yield
- Communication/Collaborations
- Future Work

Introduction

- Project Relevance
 - **2002 National Hydrogen Energy Roadmap**
 - Delivery – Efforts should focus on the development of better components for existing delivery systems, including **hydrogen sensors**...
 - Storage – Emerging hydrogen applications requires ... “smart” tanks with **integrated or embedded sensors**, ...
 - Conversion – Other key research needs include ... **more durable and lower-cost sensors**...
 - Applications – Development needs include **low-cost, fast-response, and low-power consumption sensors**...

Goals and Approach

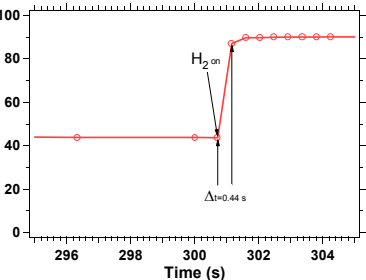
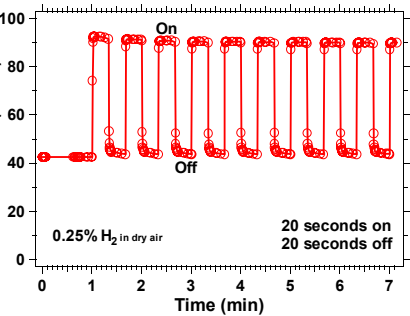
- Project Goals
 - Demonstrate the capabilities of micromachined H₂ sensors
 - Develop an understanding of their performance
 - Critically evaluate the utility and viability of this technology for life safety and processing monitoring
- Approach
 - MEMS based platform coupled with multilayer hydride films
 - Thermally controlled chemi-resistive transduction



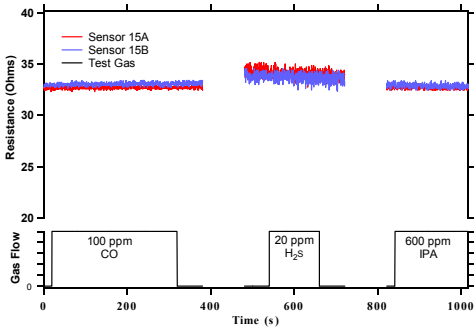
Overall Project Progression



Proof of Concept




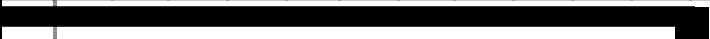












**Feasibility
Showstoppers**



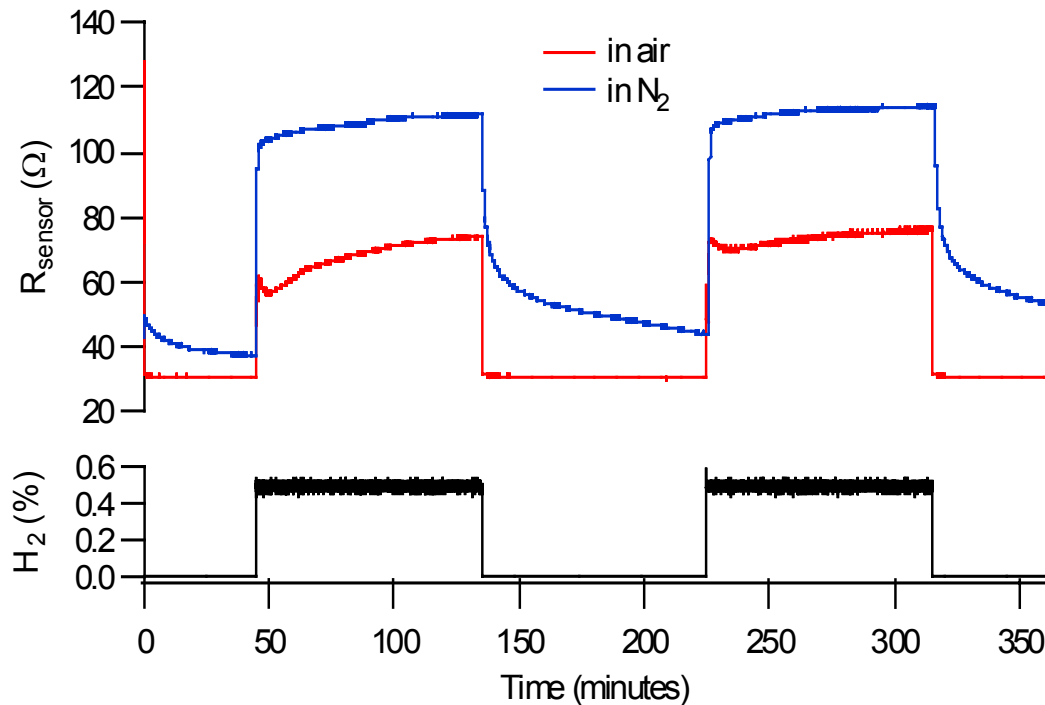
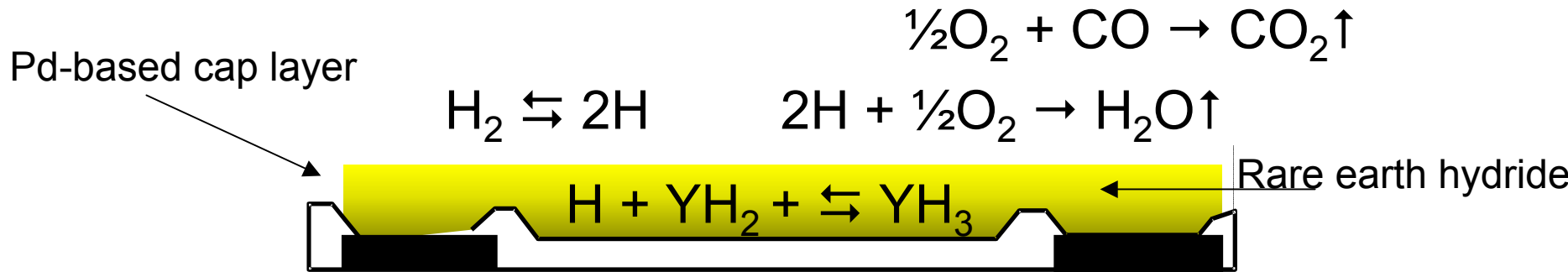
**Field Testing
Technology Extension**

**Reproducibility/Scale up
Fundamental Understanding
Systems Development
New Device Innovation**

FY 2003 Timeline and Milestones

ID		Task Name	2003												2004		
			S	O	N	D	J	F	M	A	M	J	J	A	S	O	N
1		Fabrication and Process Control															
2		Establish Process of Record															
3		Evaluate of Novel Process Step(s)															
4		Optimize Fabrication Process															
5																	
6		Sensor Characterization and Analysis															
7		Implement Quality Control Protocol															
8		Characterize Sensor Device & Materials															
9		Design Application-Specific Sensors															
10																	
11		Systems Development															
12		Finalize Package Designs															
13		Finalize Signal Conditioning Circuitry															
14																	

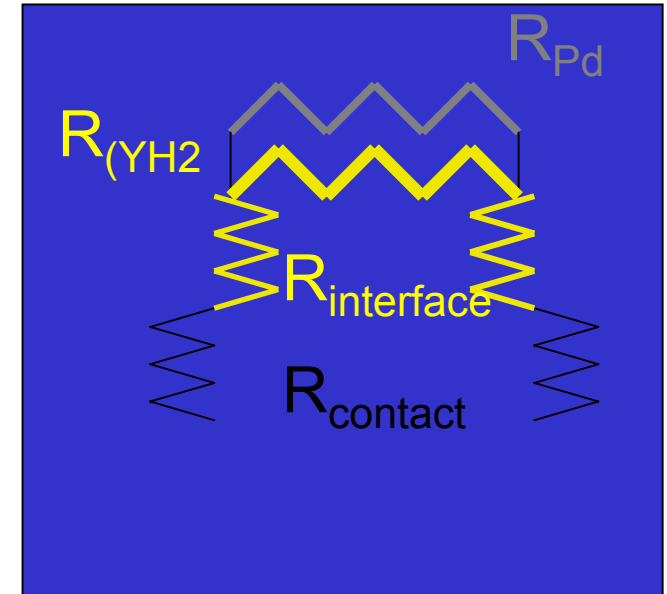
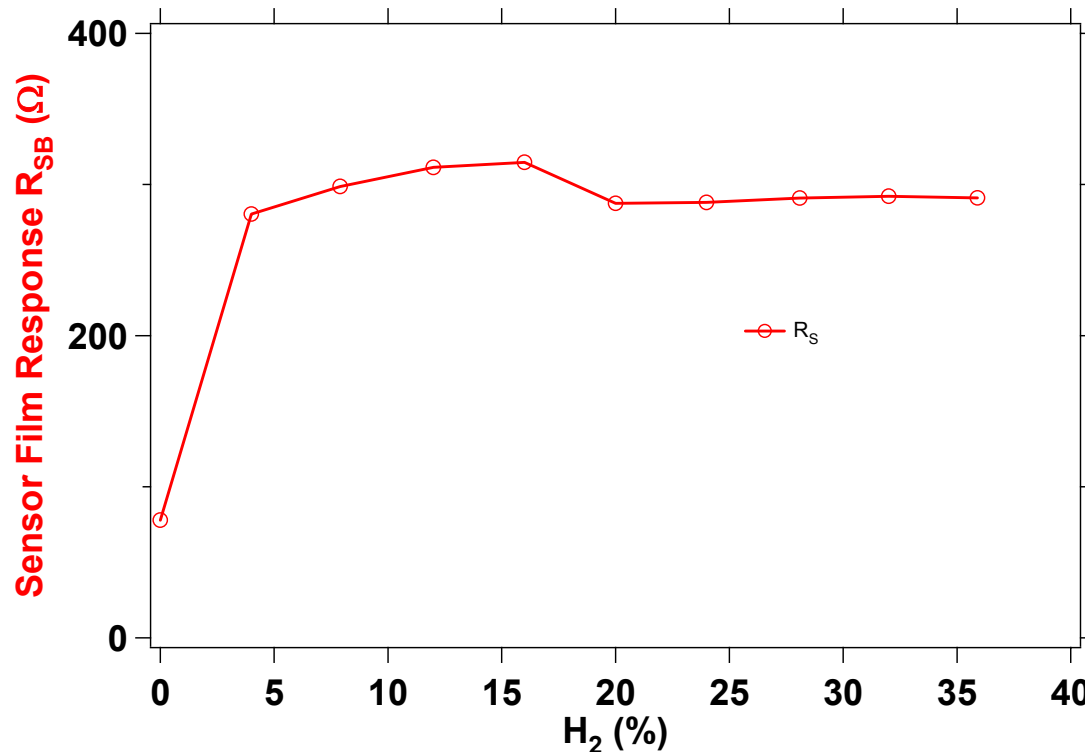
Device Model: Chemical, Electrical, Mechanical



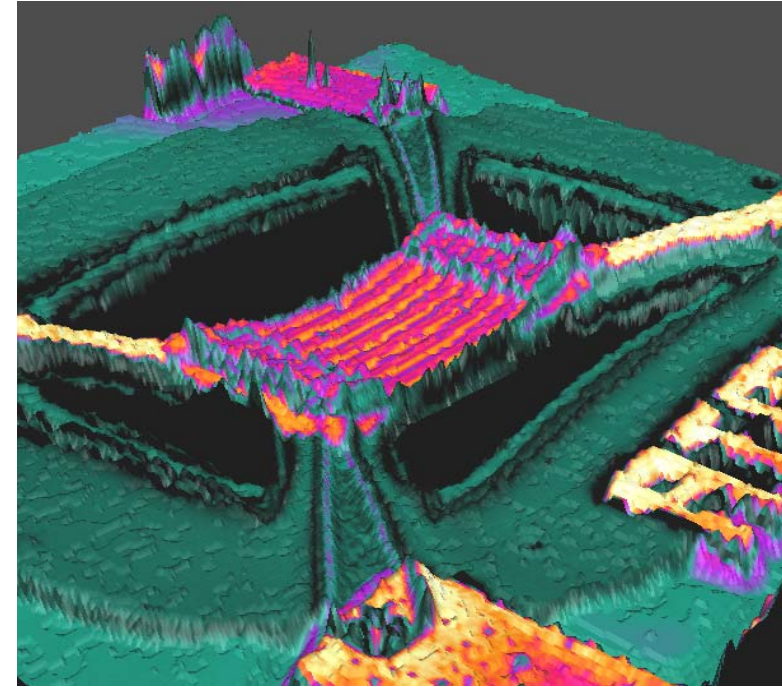
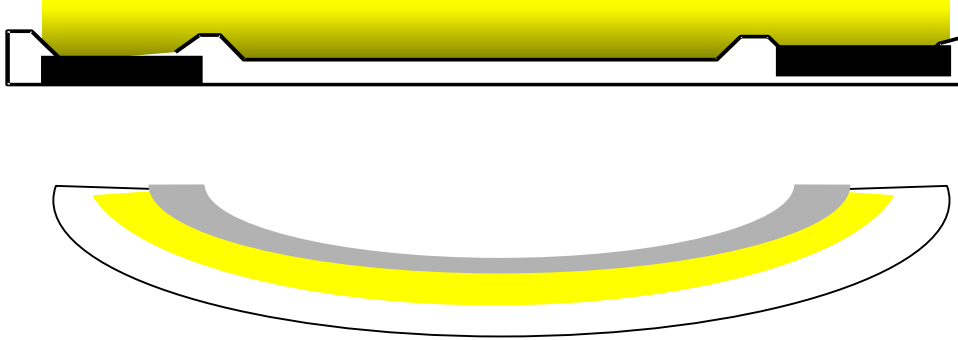
Device Model: Chemical, Electrical, Mechanical



$$R_{\text{sensor}} = 2 R_{\text{contact}} + 2 R_{\text{interface}} + R_{\text{Pd}} // R_{(\text{YH}_2 + \rightleftharpoons \text{YH}_3)}$$

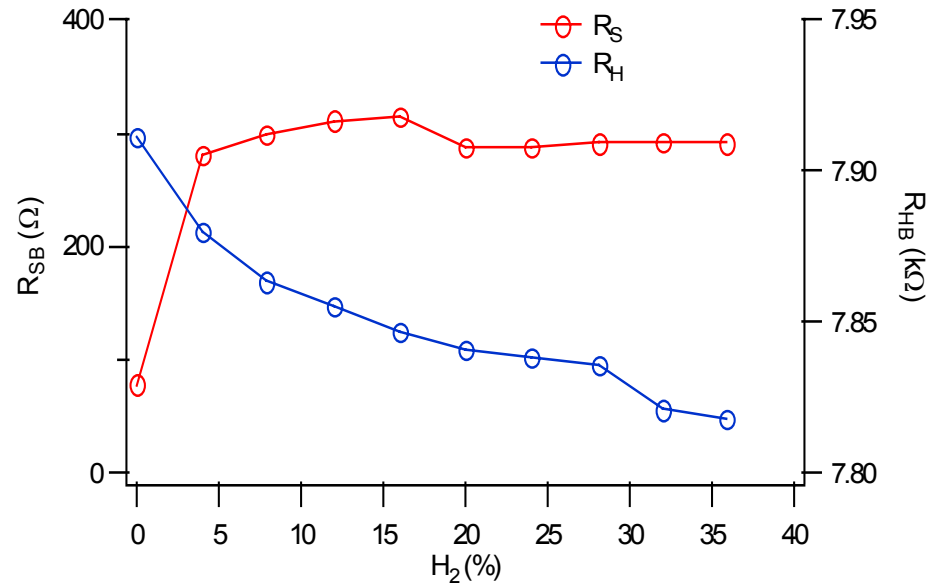
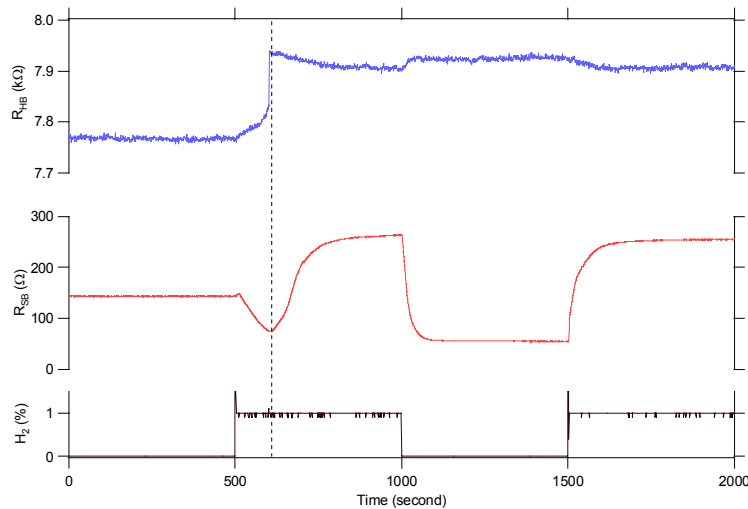


Device Model: Chemical, Electrical, Mechanical



- Volume of YHx changes by 15% from $x = 2$ to $x = 3$,
- Confocal Microscopy capability enabled measurement of bending
- Poly Silicon resistance is modulated via piezo-resistive effect
 - “Strain Gauge H₂ sensor” Pat. Pending

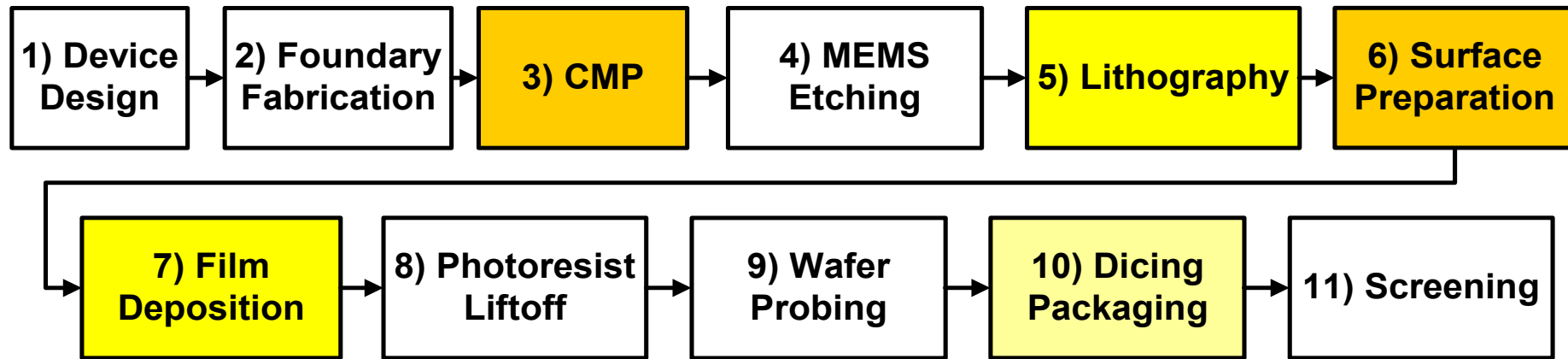
New Mechanical Transduction Data



- Potential for significant cost reduction
 - 2 wire sensor vs 4 wire sensor
 - Fewer layers
- Dual Transduction Cap

Fabrication/Process Control:

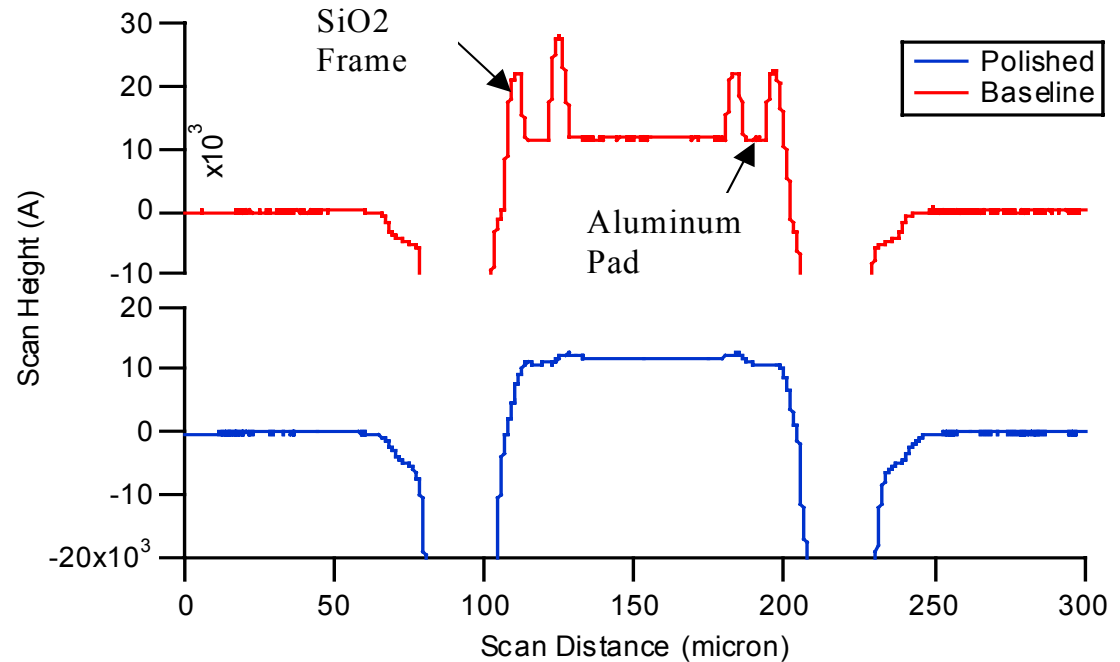
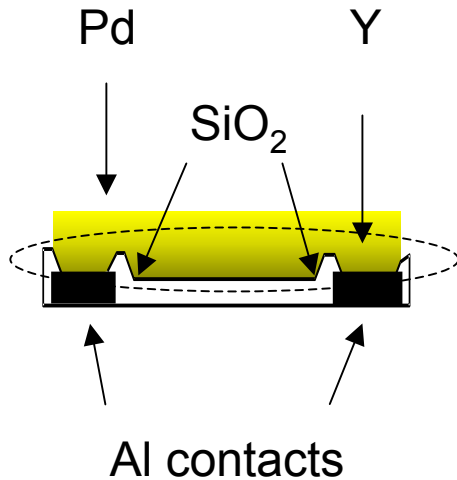
Process of Record



11 Total Process Steps with 110 Variables

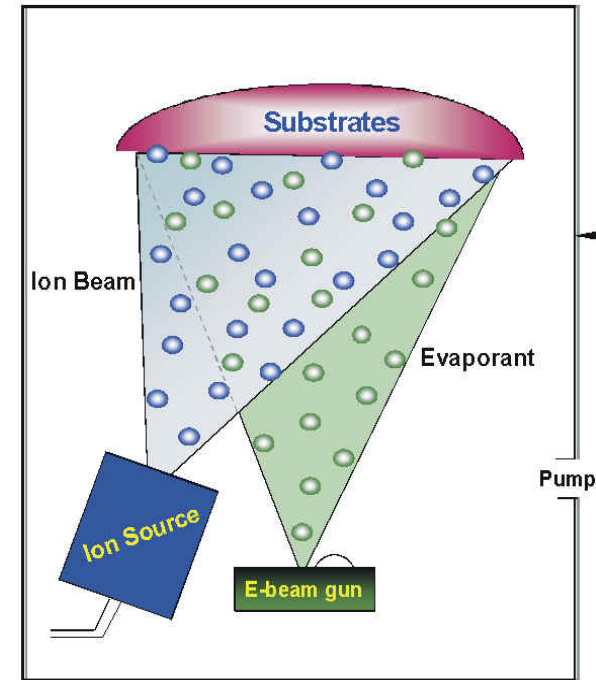
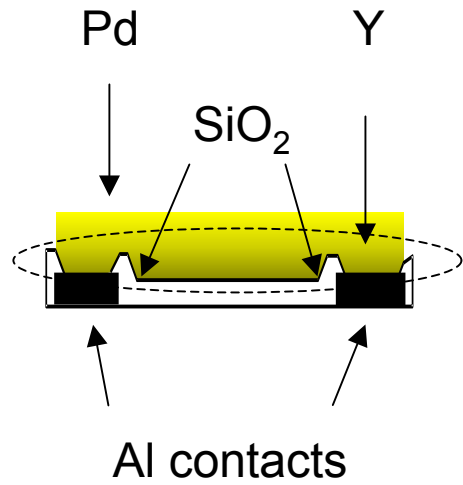
- POR forms foundation for continuous process improvement
 - Currently on V7.1
- Designed experiments and detailed process analysis focused on critical steps

Fabrication/Process Control: Chemical Mechanical Polishing



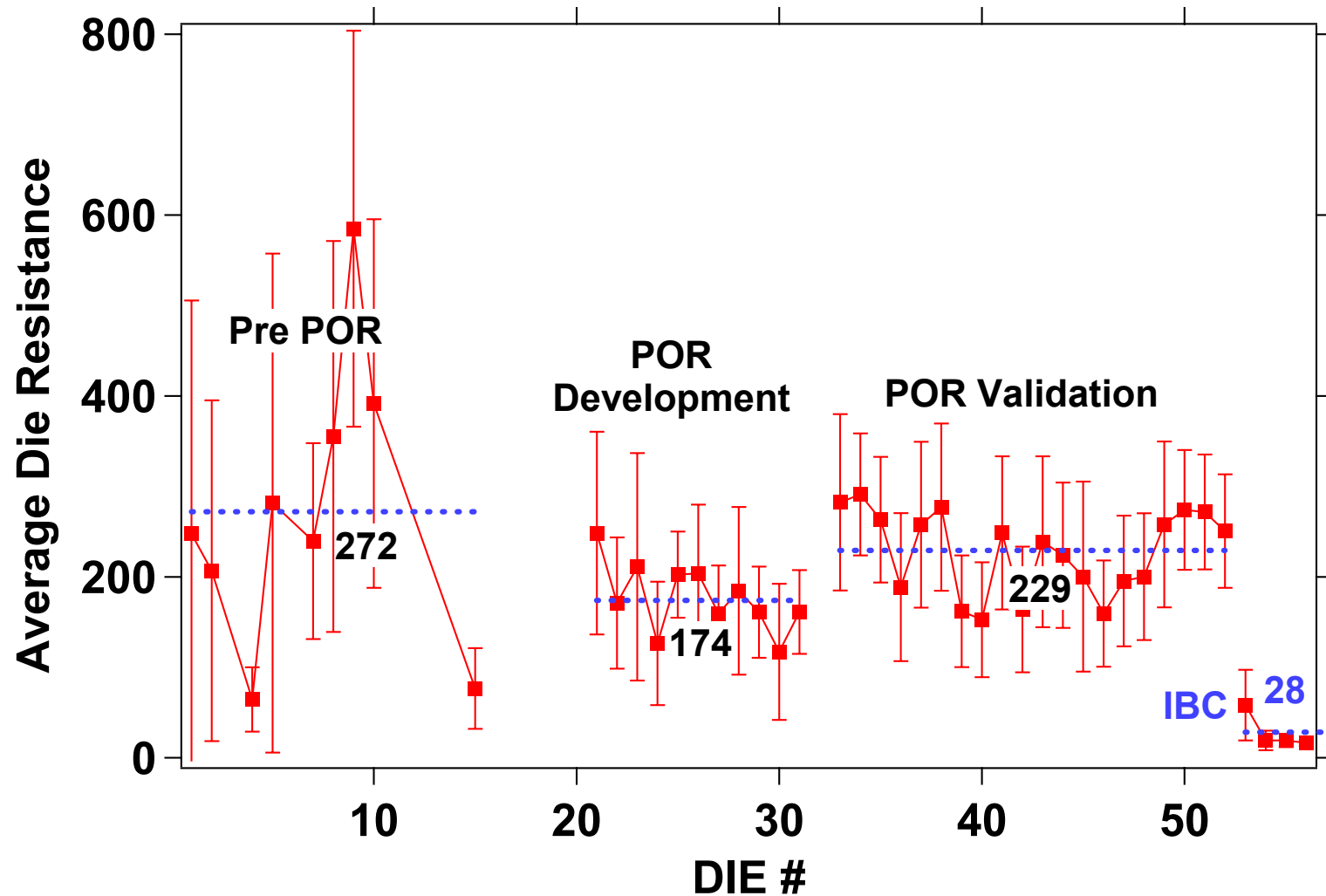
- SiO_2 frame height can be 5 to 10X functional film thickness
- Surface preparation reduces frame height and improves uniformity and yield

Fabrication/Process Control: Ion Beam Cleaning

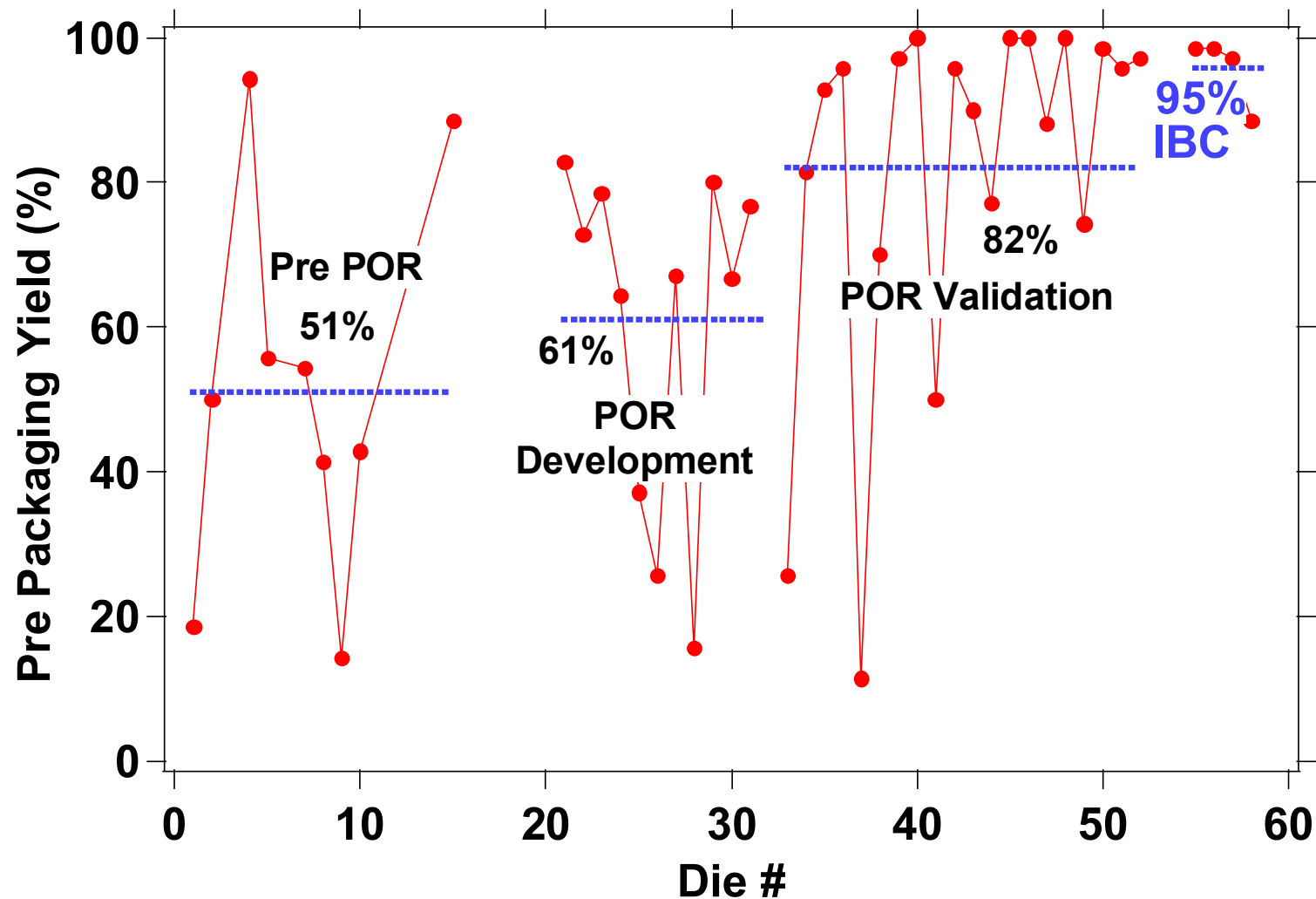


- *In situ* contact clean prevents Al contact surface re-oxidization and reduces contact resistance

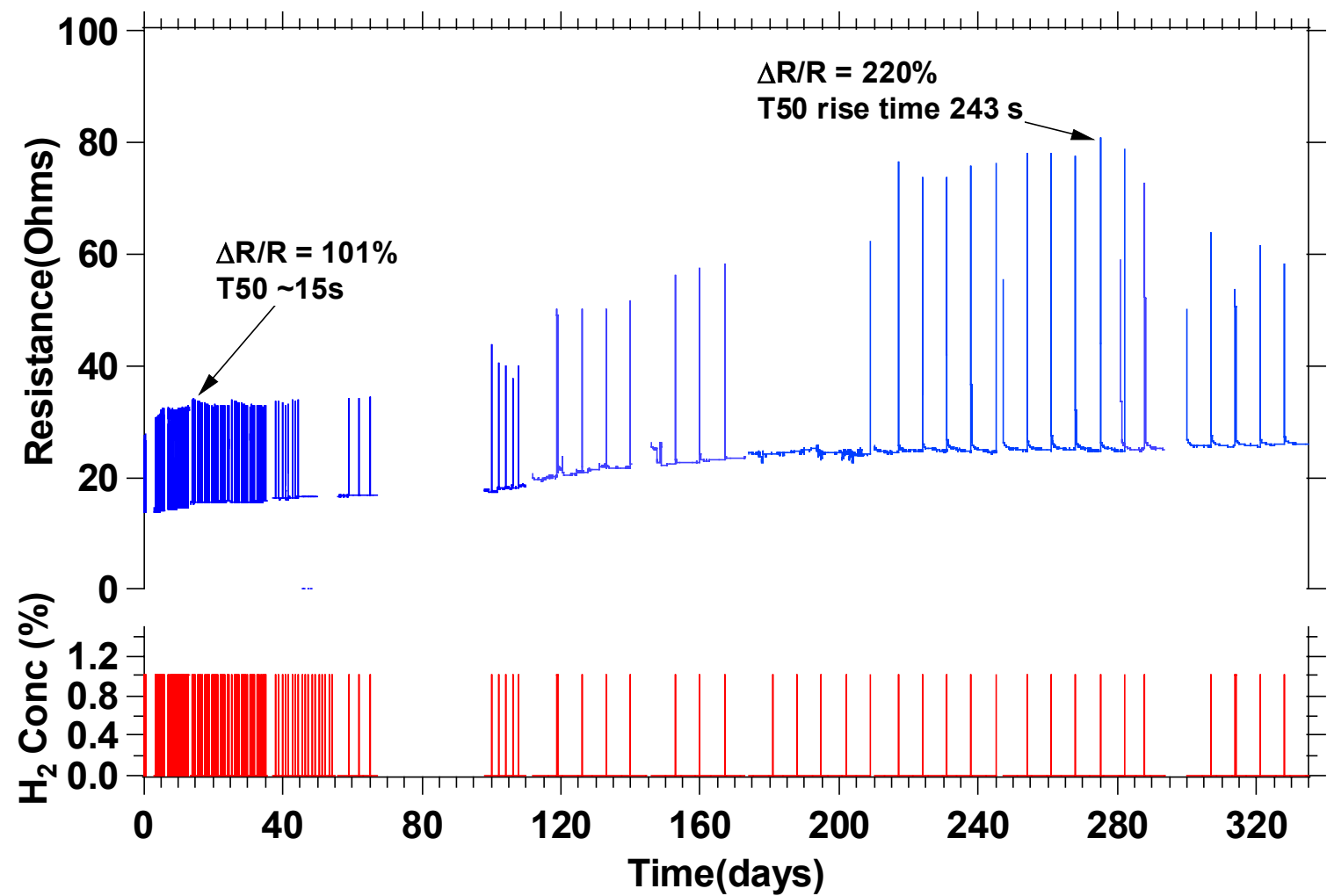
Fabrication/Process Control: Results for Die Resistances



Fabrication/Process Control: Results for Die Resistances



Systems Development: Long Term Data



Systems Development– Packaging and Signal Conditioning



- Signal Conditioning Board
 - Dual channels, PIC processor-based, on-board calibration, RS 232 and Analog out
- Self Contained Data Logging

Communication/Collaborations

- Publications and Presentations
 - “MEMS Based H₂ Gas Sensors”, submitted *Journal of Electrochemical Society*
 - “Microelectronic H₂ sensing for Emerging Sensing”, CMOC 2003
- Issued Patents
 - G. Bhandari and T.H. Baum, “Hydrogen sensor utilizing rare earth metal thin film detection element,” U.S. Patent 6,006,582 (1999).
 - F. DiMeo and G. Bhandari, “Micro-machined thin film hydrogen gas sensor, and method of making and using the same,” U.S. Patent 6,265,222 (2001).
- Allowed
 - F. DiMeo and T. Baum "Micro-Machined Thin Film Sensor Arrays For The Detection Of H₂, NH₃, And Sulfur Containing Gases, And Method Of Making And Using The Same“
- Pending
 - F. DiMeo, P. Chen, “ Rare Earth Metal Sensor”
 - I.-S. Chen and F. DiMeo, “Micro-machined piezo-resistive gas sensor, and method of making the same,”
- Collaborations
 - ATMI Life Safety Systems, ATMI GaN Products, UTC Fuel Cells

Future Work: Complete Program Milestones

- Fabrication and process control
 - Continuous improvement and refinement
- Sensor characterization
 - Re-examine optimal operation conditions based on improved fabrication techniques
 - Explore new piezo-resistive device structures
- Systems
 - Complete datalogging prototype
 - Deploy prototypes internally at ATMI

Summary/Accomplishments

- Demonstrated CMOS based MEMS based H₂ Sensors with Sub second response time
- Demonstrated hydrogen selectivity
 - no response to CO, H₂S, IPA
- Engineered sensors to operate with low power (<6mW) consumption, enabling mobile applications
- Developed an understanding of sensor behavior
 - Basic electrical device model developed
 - Demonstrated novel piezo-resistive H₂ sensor

**This technology looks promising for
meeting the sensing challenges
presented by emerging H₂ applications!**